FIFTH QUARTERLY REPORT

FOR THE PROJECT

"COMPOSITE CERAMIC SUPERCONDUCTING
WIRES FOR ELECTRIC MOTOR APPLICATIONS"

PRIME CONTRACTOR

CERAMICS PROCESS SYSTEMS CORPORATION
155 FORTUNE BOULEVARD
MILFORD, MASSACHUSETTS 01757

10 OCTOBER 1989

CERAMICS
PROCESS SYSTEMS

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CPS 89-013

DARPA ORDER NO: 9525
CONTRACT NO: N00014-88-C-0512
CONTRACT EFFECTIVE DATE: 30 JUNE 1988
CONTRACT EXPIRATION DATE: 31 MARCH 1991
PRINCIPAL INVESTIGATOR: JOHN W. HALLORAN
(508) 634-3422

Prepared for
DEFENSE ADVANCED RESEARCH PROJECTS AGENCY
1400 Wilson Boulevard
Arlington, VA 22209

OFFICE OF NAVAL RESEARCH
800 North Quincy Street
Arlington, VA 22217-5000

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*

2. REPORT DATE
10 Oct. 1989

3. REPORT TYPE AND DATES COVERED
Technical Report 1 July-30 Sept 1989

4. TITLE AND SUBTITLE
Composite Ceramic Superconducting Wires for Electric Motor Applications

5. FUNDING NUMBERS
N00014-88-C-0512

6. AUTHOR(S)
J. W. Halloran

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Ceramics Process Systems Corporation
155 Fortune Boulevard
Milford, MA 01757

8. PERFORMING ORGANIZATION REPORT NUMBER
CPS-89-013

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Defense Advanced Research Projects Agency
1400 Wilson Boulevard, Arlington, VA 22209

Office of Naval Research
800 North Quincy Street, Arlington, VA 22217-500

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES
N/A

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

This is a progress report on developing Y-123 wire for an HTSC motor. Y-123 powder lot size was increased to 10 kilograms. Spools of 125-micron diameter green fiber (50-60 vol% Y-123) are routinely produced. Wire development is focused on co-fired silver coated monofilaments. The silver coating has improved thickness and uniformity. Continuous coated green fiber has been made at lengths up to 150 feet. The major emphasis has been on continuous co-firing of wire in a reel-to-reel operation. The length of the heated zones in the furnace limits wire length, furnace modifications are being designed. Continuously sintered Ag-clad wire has been produced. The longest length without a break has been 19 feet.

Design was completed for a drum DC homopolar with an iron magnetic circuit. Parts and equipment have been ordered or are being machined. The motor will be assembled and tested with copper coils in the following quarters.

14. SUBJECT TERMS
Superconductor, ceramic, motor

15. NUMBER OF PAGES
40

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	

NSN 7540-01-280-5500

Standard Form 298 (890104 Draft)
Prescribed by ANSI Std. 239-18
298-01

COMPOSITE CERAMIC SUPERCONDUCTING WIRES FOR ELECTRIC MOTOR APPLICATIONS

EXECUTIVE SUMMARY

This report describes progress on producing Y-123 wire for an HTSC motor. The wire development activity includes synthesis of Y-123 powder, spinning polymer-containing "green fiber", heat treating the fiber to produce metallized superconducting filaments, and characterizing the electrical properties of the filaments.

Powder production facilities were upgraded to improve the jet milling facility, and to allow us to process Y-123 powder in water- and carbon dioxide-free gas. The Y-123 production lot size was increased to 10 kilograms. Quality control standards and powder specifications were improved.

We routinely produce spools of 125-micron diameter green fiber (50 vol% Y-123) in lengths up to 1000 feet as feedstock for sintering and cladding. Recently the Y-123 powder loading in the green fiber was be increased from 50 vol% to 60 vol% without loss of green fiber integrity. Higher solids loading improves burnout and sintering behavior. Efforts continue to improve the fiber spinning process and determine the influence of process variables on green fiber quality. Work on braiding multifilamentary braids continued. Eight filament braids up to 25 feet long have been produced with 125-micron fiber. Efforts continue on evaluation of alternative polymer systems.

Most wire development is now on co-fired silver coated monofilaments. Reformulation of the silver coating material has significantly improved the thickness, adherence, and uniformity of the cladding. Coating procedures were developed to produce fiber for sintering with very smooth silver coatings. Silver thickness is controllable between 20 and 100 microns, allowing variation of the metal/superconductor ratio between 1 and 8. Continuous coated fiber has been made at lengths up to 150 feet. Conditions for successful co-firing of the fiber with Ag and Ag/Pd coatings are being determined.

The major emphasis this quarter has been on continuous co-firing of silver alloy coated green fiber. Pay-out and take-up spoolers were installed in the sintering facility for reel-to-reel operation. The current process is limited by length of the heated zones, so the fiber must pass through the furnace several times for burnout, presintering, and sintering with intermediate spooling. Furnace modifications are being designed.

Continuously sintered Ag-clad wire has been produced and collected on a spool. The wire has a resistive transition in the as-sintered state and becomes fully superconducting after an oxygen anneal. Optimization of the time-temperature-atmosphere firing profile is underway, although presently constrained by intermediate spoolings. Very slow belt speeds are now being used to avoid defects associated with binder burnout. Slow speed and the need for multiple passes limits the length of sintered wire to about 25 feet. Localized defects in the silver cladding create weak spots leading to breaks. The longest length without a break has been 19 feet.

The ribbon conductor cladding module for reflow bonding has been completed. Evaluation is postponed by emphasis on the co-fired monofilamentary wire.

Electrical measurements activity this quarter was primarily for routine characterization of transport properties of sintered fibers and wires. Efforts began on several approaches to overcome weak link behavior by melt texturing.

Design was completed for the first generation HTSC motor, a drum DC homopolar with an iron magnetic circuit. Purchased parts, components, and equipment have been ordered. Major motor parts are being machined. Tests proved the performance of a brush system at liquid nitrogen temperature. The motor will be assembled and tested with copper coils in the following quarters.

The HTSC coils will be wound on a 10-inch diameter bobbin. Two coils are required with 4600 turns of monofilament wire, for a total wire length of 3000 meters per coil.

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COMPOSITE CERAMIC SUPERCONDUCTING WIRES FOR ELECTRIC MOTOR APPLICATIONS

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SECTION 1

GENERAL INTRODUCTION

This Fifth Quarterly Report covers activities during July through September 1989, on a program to develop high temperature superconducting wire by cladding $YBa_2Cu_3O_{7-x}$ (Y-123) ceramic fibers with metal, and to use this wire to build a superconducting motor. This program is being carried out by three subcontractors: an affiliate of Ceramics Process Systems, CPS Superconductor Corporation (CPSS) is charged with development of the wire; the fiber spinning technology is being developed with Albany International Research Corporation (AIResCo); and the Emerson Motor Division (EMD) of Emerson Electric is designing and will build the superconducting motor. Another contributing organization is the University of Wisconsin Applied Superconductivity Center. During this quarter there were important collaborative developments with Sandia National Laboratory, Los Alamos National Laboratory, and Arthur D. Little Inc.

The status of the program is compared with individual tasks of the revised Statement of Work in Figures 1.1.1, 1.1.2, and 1.1.3, which show timelines for the major fiber and wire development tasks.

The following sections describe in detail the progress on the wire manufacturing task and the motor design task. Section 2 covers all aspects of the wire manufacturing development, including the fiber spinning work con-

ducted at Albany International Research Corporation in Mansfield, Massachusetts, and the Y-123 powder production, sintering, cladding, and characterization work conducted at CPS Superconductor in Milford, Massachusetts. Section 3 outlines the HTSC motor design activities at Emerson Motor Division in St. Louis, Missouri.

```
                  1988        1989                    1990                    1991
                  J A S O N D J F M A M J J A S O N D J F M A M J J A S O N D J F M
                   1  |  2  |  3  |  4  |  5  |  6  |  7  |  8  |  9  | 10  | 11  |
                                                  .
                                                  .
                  Rh-P dry                        .
                  spin fiber                      .
                  [======---]                     .
                      CPSS dry spin fiber
FIBER                 [================------
DEVLP                                             .
                  BaTiO3 melt spinning
                  [=====================-------
                                                  .
                           Y-123 melt spinning
                        [======================================]
                                                  .
                                         Green  . fiber braiding
                                        [======================]
                                                  .
                                                  .
                   1  |  2  |  3  |  4  |  5  |  6  |  7  |  8  |  9  | 10  | 11  |
```

Figure 1.1.1   Revised Project Schedule for Fiber

```
                1988       1989                    1990                    1991
                J A S O N D J F M A M J J A S O N D J F M A M J J A S O N D J F M
                 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 |
                                                 .
                Lab sinter                       .
                [=======================--       .
                                                 .
                      RTC furnace
                      [==============
WIRE                                             .
                                                 .    Ribbon wire
DEVLP                                            . ===================================
                                            ↑ POSTPONE
                   Cladding module            RIBBON WIRE
                   Design and build              .
                   [======================
                                                 .
                Silver coat and co-fire
                [==============================---------------------
                                                 .
                       Silver monofil wire
                                 [==========================================
                                                 .
                 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 |
```

Figure 1.1.2 Revised Project Schedule for Wire Tasks. Note that Ribbon Wire had been postponed in favor of monofil wire

```
                 1988        1989                    1990                    1991
                 J A S O N D J F M A M J J A S O N D J F M A M J J A S O N D J F M
                   1  |  2  |  3  |  4  |  5  |  6  |  7  |  8  |  9  | 10  | 11  |
MOTOR                  application           .
DELVP                    study               .
                       [==========]          .
                                             .
                 define properties        build test equip
                 [=====================]  [================]
                                             .
                                          test short
                                           wire       test coils
                                          [=======][====================]
                                             .
                                       design   fabr     test
                                       motor    motor    motor
                                  [==============][========][=================]
                                             .
                                          advanced motor design   build
                                             . [==============]===============]
                                             .
                   1  |  2  |  3  |  4  |  5  |  6  |  7  |  8  |  9  | 10  | 11  |
```

Figure 1.1.3   Project Schedule for HTSC Motor Task

SECTION 2

WIRE FABRICATION

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2.1 Introduction and General Comments

The emphasis this quarter was on continuous sintering of silver-clad monofilamentary wire. Our major goal was reel-to-reel sintering to produce prototype spools of wire, so activities focused on overcoming practical problems uncovered in the continuous sintering process. Three improvements were necessary to achieve prototype spools. The sintered wire needs a 20-50 micron metallurgically sound cladding to survive handling and spooling, so the thickness and uniformity of the powder metal (PM) silver alloy coating on the green fiber needed to be improved. Binder burnout proved to be a more difficult step with the thicker PM silver coatings, and some experimentation was required to find successful conditions. Finally, sintering conditions had to be established to successfully densify the Y-123 substrate and the PM silver alloy coating in a single co-firing step.

Other activities were related to improving Y-123 powder production and the melt spinning process for green fiber, fiber braiding for multifilamentary wires, and several explorations of melt texturing processes.

The detailed progress reports for each of the four wire process steps are presented in Sections 2.2 through 2.5.

2.2 Fiber Preparation

2.2.1 Introduction

During this quarter the major activities in fiber preparation were in support of continuous firing. This involved producing larger quantities of the "standard" 125-micron diameter high density polyethylene (HDPE)-based green fiber with 50 vol% Y-123 powder, and examining firing problems which were believed to be related to the green fiber. Efforts continued on improving green fiber production, evaluating alternative polymer systems, and production of green fiber braids.

2.2.2 Powder production

A number of improvements were made in the process for producing the 1.6-micron diameter Y-123 powder. Lot size was increased to 10 kilograms raw batch. Powder production facilities were upgraded to improve jet millin by processing in nitrogen, and calcination by processing in air thoroughly scrubbed of carbon dioxide (≤2 ppm) and water (-100°F dew point). Initial calcination data suggests that it may be possible to reduce calcination temperature, which would permit production of even finer Y-123 powder. Quality control standards and powder specifications were improved during this quarter. About twenty lots of powder were supplied to support the fiber experiments.

2.2.3 Green fiber spinning

Much of the effort this quarter was directed at producing a consistent green fiber for prototype wire. We routinely produced spools of 125-micron diameter green fiber (50 vol% Y-123) in lengths up to 1000 feet.

There was a continuing effort to determine the effect of the spinnerette die and process variables such as compounding conditions, extrusion rate, temperature and pressure on the quality and behavior of the green fiber. A number of experiments were conducted in an attempt to track down the cause of certain defects which occasionally arise during binder burnout. For example, an occasional batch of green fiber will curl during burnout for reasons which have not yet been determined.

We learned that the Y-123 powder loading in the green fiber can be increased from 50 vol% to 60 vol%. The 60% fiber has similar processing characteristics and green fiber properties, but the higher solids loading should improve burnout and sintering. This fiber is now being evaluated.

We continued to explore alternative polymer systems which could be better than the current high density polyethylene blend. This quarter we evaluated three different grades of HDPE which differ from the present grade by having either (1) higher molecular weight, (2) lower molecular weight, or (3) a bimodal molecular weight distribution. All of these systems produced poorer quality green fibers than the standard grade of HDPE. We also evaluated several other types of polymer, including polymethyl-pentene, polyethyl terephthalate, and elastomeric polybutyl terephthalate. All of these produced inferior green fiber. As the quarter was ending, however, we had very encouraging results by melt spinning barium titanate green fibers using a polyvinyl butyral (PVB) resin. The PVB fiber was very easily spun, quite strong, and exceptionally flexible. PVB is commonly used as a binder in oxide ceramics, so will probably have acceptable burnout behavior. We intend to explore Y-123 fibers with PVB early in the next quarter.

2.2.4 Braiding of Y-123 green fibers

More progress was made on braiding green fibers for use in multi-filamentary wire. Previously only large diameter 250-micron green fiber could be braided. Attempts to braid the standard 125-micron fibers failed as the fibers frequently broke during the process. The braiding equipment was modified to accommodated the low strength of the 125-micron green fiber. The modified equipment was able to braid 175- and 125-micron fiber into an eight filament braid which, although it had many breaks and defects, had sections as long as 30 cm without any filament breaks. The total length of the braid was about ten meters. These braids were used to evaluate sintering and to fabricate prototype samples of multifilamentary wire.

These experiments were tubular or round cross sectional braids. Flat braids are desired for flexible ribbon wire. A braiding machine capable of producing flat braids became available at the end of this quarter, and will be used for future experiments.

2.3 Sintering and Cladding

All work is now on co-fired silver alloy coated monofilaments. These are made by coating the green Y-123 fiber with a powder metal silver alloy coating, and "co-firing" to simultaneously densify the Y-123 and the PM silver alloy. The tasks involve developing the PM coating, adjusting for the more difficult burnout, and achieving a successful co-fire.

2.3.1 Cladding and co-firing

A significant reformulation of the PM silver coating material improved thickness, smoothness, and uniformity of the cladding substantially. Figure 2.3.1 shows a 125-micron green fiber (on left) and a similar fiber with a 40-micron thick green PM silver coating (on right), illustrating the uniform thickness and smooth surface. The silver thickness can be easily adjusted between 20 and 100 microns, allowing variation of the metal/superconductor ratio between 1 and 8. The thickness is controlled by adjusting the applied thickness per coat and the number of repeated coatings. The use of repeated coatings also allows us to change the composition of each layer, providing an extra degree of freedom which we a; beginning to explore. The improvement in uniformity arises primarily from an improved coating material which has better dispersion of the metal powder and rheology. Process improvements have also been made to improve concentricity, insure more constant fiber motion, and eliminate vibrations. Continuous coated fiber has been be made at lengths up to 150 feet.

The adhesion of the PM silver coating to the green fiber has been improved, but is still not entirely satisfactory. One problem is the fact that the current green fiber is made with polyethylene, a polymer whose waxy

surface is a notoriously difficult substrate for bonding. A second problem arises when the coated green fiber is stressed. The present PM silver coating is more brittle than the green fiber. Any stresses which elastically stretch the fiber can cause cracking or delamination of the coating. Both these problems are being addressed.

Co-firing introduces other challenges. After high temperature exposure, the metal usually adheres tenaciously to the Y-123 filament. However, if the sintering condit·ˇns are incorrect, or if the green coating has been locally damaged, the Y-123 filament may shrink away from the cladding, creating a local delamination defect. Or, if the metal cladding tends to shrink more than the Y-123 substrate, the cladding may tear locally. Tears and delaminations are the most common cladding defects observed in the prototype wire, occurring to a greater or lesser extent in all specimens made to date. Often wire breaks occur at these defects. They are becoming less frequent as we gain more experience and learn to ¬ore carefully handle and sinter the coated fiber.

Conditions for successful densification of the Y-123 during co-firing of the fiber with Ag and Ag/Pd coatings are being determined. Silver-palladium alloys were originally chosen to raise the melting point of the cladding above the sintering temperature of the stoichiometric Y-123 fiber. With Ag-18Pd coatings we have been able to achieve high density Y-123 after continuous sintering at a peak temperature of 965°C in air. (Attempts to sinter above 970°C have been unsuccessful, apparently due to a reaction between Pd and Y-123 which degrades the microstructure of the superconductor.) The 965°C/air sintered Y-123 has good electrical properties, but unsatisfactory mechanical properties. The co-fired Ag-Pd cladding is brittle and

does not strengthen the wire. Figure 2.3.2 is a fracture surface of a 945°C sintered wire with a 60-micron thick Ag-Pd cladding. The Y-123 (on right) has satisfactory density, but the cladding (left) exhibits a brittle fracture surface. Note that the Ag-Pd alloy system is a simple substitutional FCC solid solution, so should be quite ductile. We have not yet identified the cause of the embrittlement of this alloy.

Most of our recent work has been with pure silver cladding. Silver is much less expensive than palladium, and high quality silver powders are more readily available. The silver claddings are strongly adherent, very ductile, and of good metallurgical quality. The disadvantage of silver is that its melting point is very close to the sintering range of our stoichiometric Y-123. We find that our silver coatings melt at temperatures greater than about 930°C in air, due to formation of the Ag-Ag_2O eutectic.

We have adopted two approaches to co-fire with silver. By sintering at 925°C we are able to achieve densities estimated around 90% of theoretical. Figure 2.3.3 shows a polished section from a 12-foot continuous wire sintered in air at 925°C. The superconducting core has some residual porosity. The silver cladding is dense, adherent, and metallurgically sound. Upon fracture, the silver undergoes extensive plastic deformation and exhibits a fracture pattern typical of ductile rupture.

The second approach to silver co-firing is to suppress the Ag-Ag_2O reaction by sintering in nitrogen. By flowing nitrogen into the RTC furnace the oxygen content of the exhaust gas can be kept below 300 ppm, which is sufficient to prevent silver melting below 950°C. The lower oxygen partial pressure also reduces the sintering temperature of the Y-123. Acceptable sintered densities have been achieved by nitrogen co-firing at 945°C. However

process control has been difficult, and occasionally sections of wire have had melted silver cladding. We also find that nitrogen sintering has a smaller processing window. For example, apparently minor changes in the binder removal schedule can lead to major phase decomposition reactions at the sintering temperature. It seems that the Y-123 is much less tolerant of carbonaceous binder residue during nitrogen sintering. We have had successful Ag/nitrogen co-firings, but more process development is needed. We are starting to explore oxygen/nitrogen mixtures in the hope that the carbon removal can be accomplished at oxygen contents still low enough to avoid the silver oxide reaction. It is also possible to sinter in a 925°C/air zone, followed by a final densification in a 945°C zone, although this is inconvenient with the present belt furnace.

After the 925°C/air co-fire, the wire is weakly superconducting, exhibiting a fairly broad resistive transition at 85K. An oxygen anneal for 10 hours at 550°C sharpens the resistive transition and provides a transport critical current in the lower range of our "typical" filaments. The 945°C-/nitrogen co-fired wire, as expected, is not superconducting in the as-fired case. The same oxygen anneal restores the typical superconducting properties.

2.3.2 Continuous sintering

Our main activity was to develop methods for continuous co-firing of silver alloy coated green fiber. All heat treatment is now done in the Radiant Technology Corporation (RTC) belt furnace. As described in the previous report, this furnace has a 6-inch wide metal mesh belt to move ware through a 30-inch hot section and a 45-inch cooling section. The hot section is divided into three zones with independent control of temperature and

atmosphere. The heating elements are quartz lamps, providing primarily radiant heat transfer. To configure the RTC as a fiber sintering furnace, a Nextel belt has been installed to cover the metal mesh belt. Pay-out and take-up spoolers were installed for reel-to-reel operation.

The RTC was acquired as an experimental lab-scale continuous sintering furnace, and so has several shortcomings. The length of the heated zones is only 24 inches, so very slow belt speeds are required to accumulate residence time in the hot zone. We cannot do both binder burnout and sintering in one pass, since it is not possible to maintain the third zone at 925°C for sintering, while still controlling the first zone below 300°C for the critical early stages of binder burnout. To overcome these problems, the fiber must pass through the furnace several times for burnout, presintering, and sintering with intermediate spooling. The requirement of intermediate spooling unfortunately constrains the firing process, since the fiber exiting after each pass must survive the handling. Consequently our multi-pass continuous sintering is done with a schedule which accommodates the spoolings, rather than optimizes the burnout and sintering.

Furnace modifications are being designed to eliminate these problems. A new heating section will be built onto the furnace for binder burnout. This section will be a series of heated zones long enough to allow one-pass sintering at faster rates. The zones will be heated using conventional resistance heaters rather than radiant heaters. Resistance heaters are better suited for low temperature binder burnout. The modified RTC will be used to prepare second generation continuous wire, and provide data for design of a pilot scale furnace.

Very slow belt speeds are now being used to avoid defects associated with binder burnout. In some cases, belt speed through the critical burnout range is as slow as 0.5 inch/minute. The slow speed and the need for multiple passes limits the length of sintered wire to about 25 feet. The longest length without a break has been 19 feet. Localized defects in the silver cladding create weak spots, leading to breaks which usually occur during spooling.

The time-temperature-atmosphere firing profile was explored in an extensive series of experiments, although the range of variables was constrained by intermediate spoolings. We are now engaged in short length (one meter) experiments to improve the schedule without regard for the need for intermediate spooling. The improved schedule will be implemented for continuous co-firing later in the next quarter when the furnace modifications are complete.

2.3.3 Multifilamentary ribbon conductor

The ribbon conductor cladding module, for reflow bonding of Y-123 filaments to soldered copper strips, has been completed. Evaluation is postponed by emphasis on the co-fired monofilamentary wire.

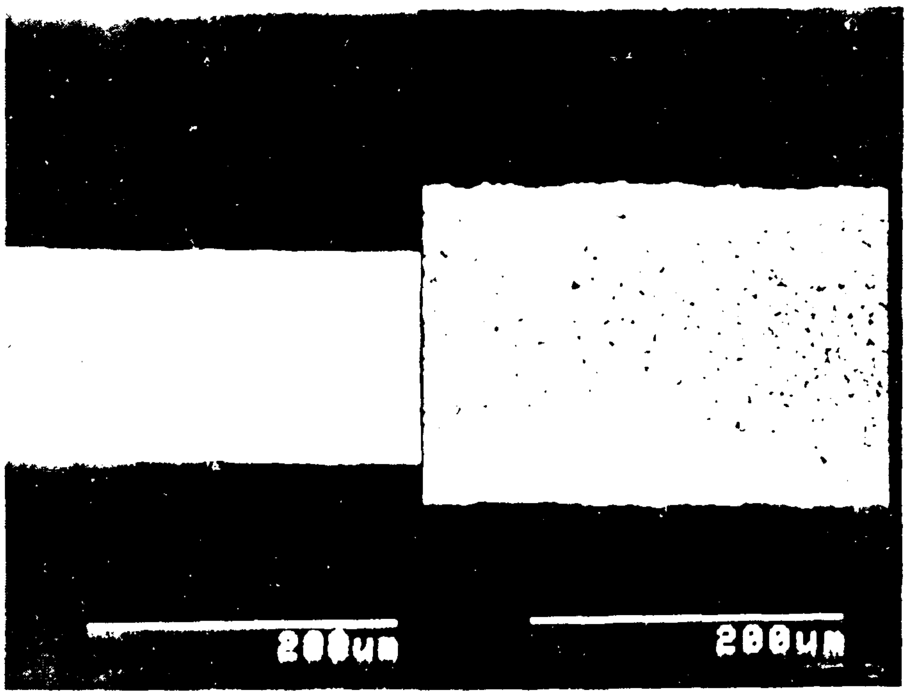


Figure 2.3.1 Green Fiber Before and After Coating With PM Silver Green Cladding

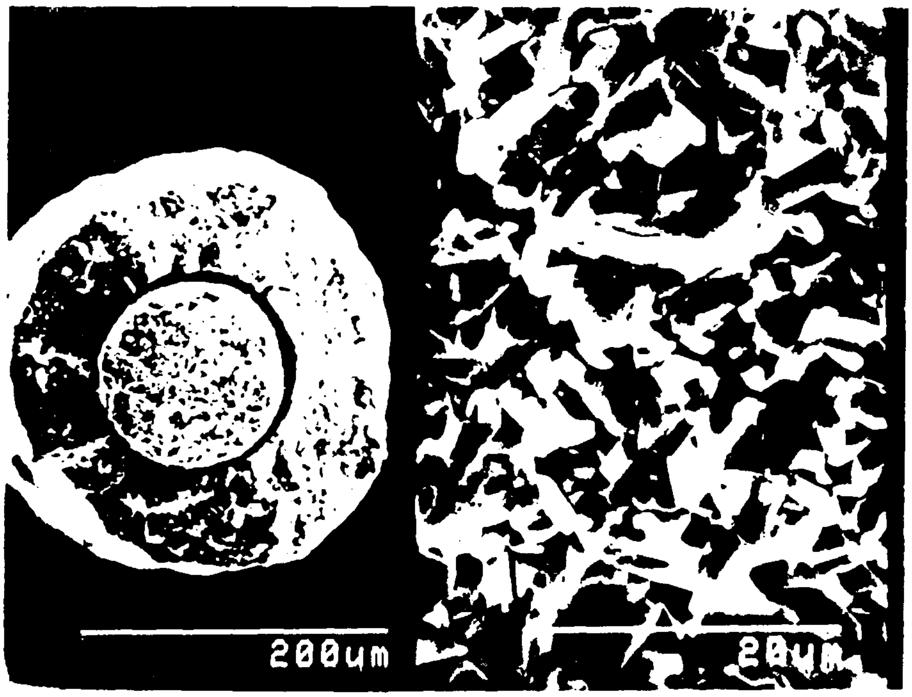


Figure 2.3.2 Silver-Palladium Clad Y-123 Wire After Continuous Sintering At 945°C in Air
Left: Fracture Surface Showing Brittle Fracture of Cladding
Right: Microstructure of Y-123 Core

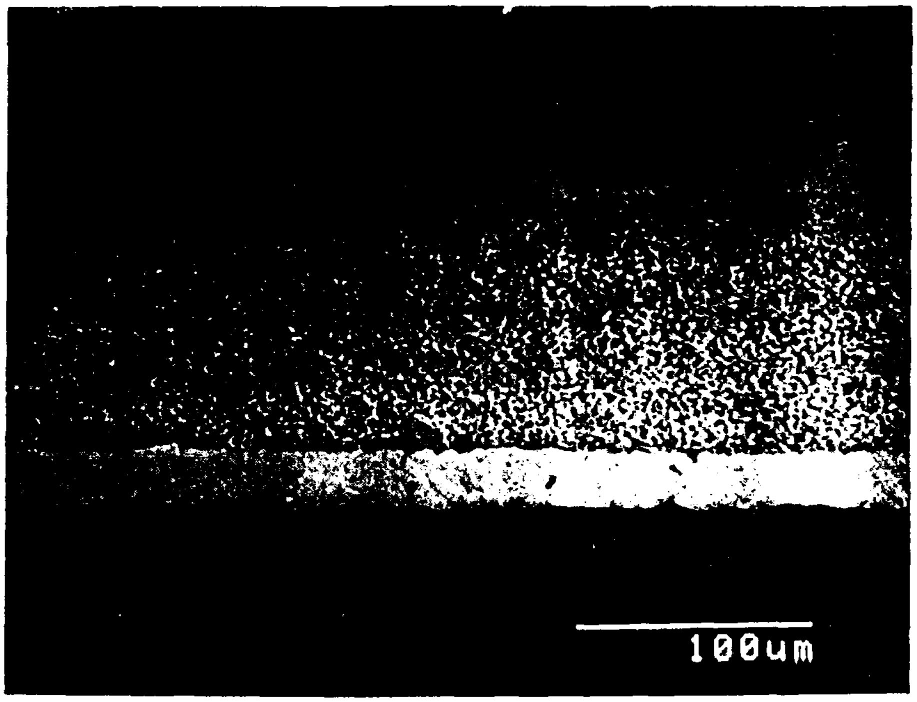


Figure 2.3.3 Polished Section of Silver Clad Wire Continuously Sintered at 925°C in Air

2.4 Electrical Characterization

Activities in electrical characterization were mostly routine characterization of continuously sintered wire before and after a post-sintering oxygen anneal. The continuously sintered wire is superconducting, but is not yet as good as the short filaments produced previously with the optimized zone sintering method. Figure 2.4.1 shows the resistive transition of a continuously sintered 925°C/air silver clad wire in its as-sintered condition. We were surprised to find a broad transition with an 88K onset which does not quite go to zero resistance at 77K. This is surprising since the as-sintered wire was cooled at a rapid rate around 100°C/minute as it passed out of the heated zone, and experienced less than 10 minutes in the 600-500°C range typically used for oxygen annealing. This data is from a sample of the wire shown in Figure 2.3.3, which had a 35-micron thick dense silver cladding and a 90+% dense Y-123 core. The porosity in the core must contribute to the rapid oxygen pickup during the cooling. It is significant that the silver cladding was not a barrier to oxygen transport. After a conventional 10-hour 550°C anneal in flowing oxygen, this wire has a sharp resistive transition, as shown in Figure 2.4.1.

The 945°C/nitrogen sintered wires are, of course, not superconducting in the as-sintered state. The conventional oxygen anneal restores superconductivity. A resistive transition for a sample of this wires is shown in Figure 2.4.2. Notice that at this stage of development, the resistive transition is not sharp. Onset is delayed to 89K, and a tail persists down to 83K. We expect that improvements in sintering and annealing will allow us to sharpen the transition.

Relatively few critical current density measurements were performed during this quarter. Many of the early continuously sintered wires had poor resistive transitions and very low J_c values. The highest values obtained in the recent continuous wire are 100-200 A/cm^2. These values are lower than the 1500-2400 A/cm^2 values previously obtained with short zone sintered bare filaments. This reflects the sub-optimal microstructure of present co-fired wire.

A systematic annealing study is underway, relating transport and magnetic susceptibility to the temperature-time schedule of the oxygen anneal. Results of this work will be available next quarter.

2.5 Melt Processing of HTSC Wires

It is unlikely that the weak link problem will be overcome with any sintered polycrystalline HTSC material. Grain orientation, while perhaps necessary for high J_c, is clearly not sufficient. Several groups have reported textured polycrystalline ceramics with very high degrees of orientation which still exhibit weak link behavior, albeit with improved values of self-field critical current density. Melt texturing is the only demonstrated method for producing strongly linked bulk materials.

We have begun to explore melt processing methods which could be employed to produce wire. Three concepts are now being evaluated. The key feature of each concept is compatibility with wire processing. These are: (1) laser float zone growth; (2) rapid thermal anneals; and (3) melt/quench powder.

Preliminary exploration of laser float zone (LFZ) processing is being done in collaboration with Dr. E. Peters of Arthur D. Little Inc. Using

a polycrystalline Y-123 filament supplied by CPSS, they have demonstrated laser melting and processing with a floating liquid zone. A post-anneal restores the Y-123 phase. Initial work is very encouraging and will be pursued. Laser float zone processing is attractive because it obviates the need for a container for the melt (since it is held in the "floating" molten zone by surface tension), and it is compatible with filamentary feedstock. The existing apparatus at ADL accepts a 10-20 cm long feed rod. This process is being explored as a continuous crystal growth/melt processing method, since the method can be adapted to use continuous feed filaments. The concept is to introduce laser float zone processing as a post-sintering treatment for Y-123 or BiSCCO fibers. For both of these systems there is a high probability of success for producing high critical current density, since LFZ is a true crystal growth technique. A disadvantage is the slow growth rates inevitably associated with crystal growth. Thus the process may prove to be feasible, but impractical for manufacturing.

The rapid thermal anneal (RTA) explorations is part of a vigorous collaborative program with David Ginley at Sandia National Laboratory. He is exploring the application to HTSC materials of very short duration anneals, typically 1-2 seconds at high temperature. Local melting and resolidification of the Y-123 phase occurs in RTA. Some specimens have local regions with a "melt-textured" microstructure, and it seems possible that RTA could be used as an ultra-rapid melt texturing process.

The RTA fibers have remarkable properties. The Y-123 undergoes partial melting, densification, and microstructure development while maintaining its shape. In fact, braided fibers retain the individual filament shape during RTA. Figure 2.5.1 shows an RTA processed seven filament braid

made from 150-micron diameter Y-123 fibers. This was produced with a treatment in which the green braid (after a conventional 600° binder burnout) was heated 1-second at 1025°C.

A more interesting phenomenon is zero-resistance superconductivity in RTA Y-123 filaments without the need for an oxygen post-anneal. For example, we have produced as-sintered superconducting Y-123 in RTA anneals in which we heat to high temperature in 4 seconds, hold at peak temperature 1-4 seconds, and quench to 600°C in 96 seconds. The fiber is cooled to room temperature within 3 minutes after starting the cycle. The resistive transition for the RTA braid is shown in Figure 2.5.2.

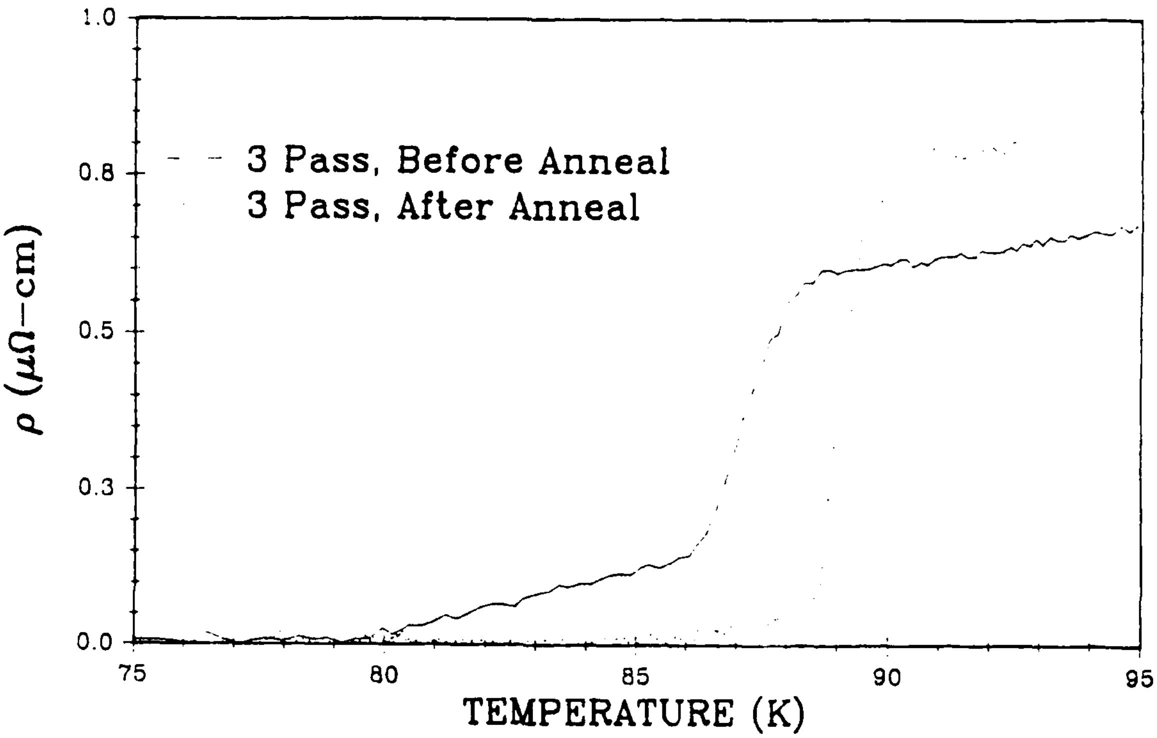
In addition to RTA of green fibers, we have explored RTA of sintered filaments. :gure 2.5.3 shows a transition to zero resistance (without oxygen anneal) after RTA processing of a pre-sintered Y-123 filament. Shown for comparison is the resistivity trace for a similar as-sintered filament which did not receive an RTA treatment. There is no hint of a resistive transition down to about 83K.

So far the RTA fibers have had disappointingly low critical current densities. However our research is still in an early stage, and the possibility of ultra-rapid melt processing justifies further exploration.

The third method being explored involves melt/quenched Y-123 powder. It is predicated on observations made by Sawano et al. at Nippon Steel[1] about the role of the quenching process in their Quench and Melt Growth (QMG)[2] method. Nippon Steel claims that their QMG processed Y-123 exhibits 10,000 A/cm^2 at 1 T from Bean Model magnetization calculations, and apparent strong flux pinning, again inferred from magnetization data. The QMG process is an elaborate sequence of melting Y-123 compositions, quenching the

melt to form a slab, followed by three annealing processes to :1) nucleate yttria grains, 2) form 211 stringers by reaction, in a gradient, between yttria and the melt, 3) peritectic reaction to form Y-123 with textured microstructure.

Sawano et al. believe that one of the key steps is the nucleation of yttria following the first melt/quench. If this is true, it might be possible to form the melt/quenched slab into fibers before the subsequent anneals, and carry out the texturing process with the material in the form of a continuous fiber. This could be done by crushing and milling the quenched melt, and spinning fibers from the resulting powder. Initial studies of melting behavior have been performed, and a future program has been identified to explore these concepts.



Cofired fiber, fired in air. 1st pass to 270°C, 2nd pass to 920°C, 3rd pass to 925°C.

Figure 2.4.1 Resistive Transition of a Silver Clad Wire Continuously sintered at 925°C in Air in the As-Sintered State and After a Post Anneal in Oxygen

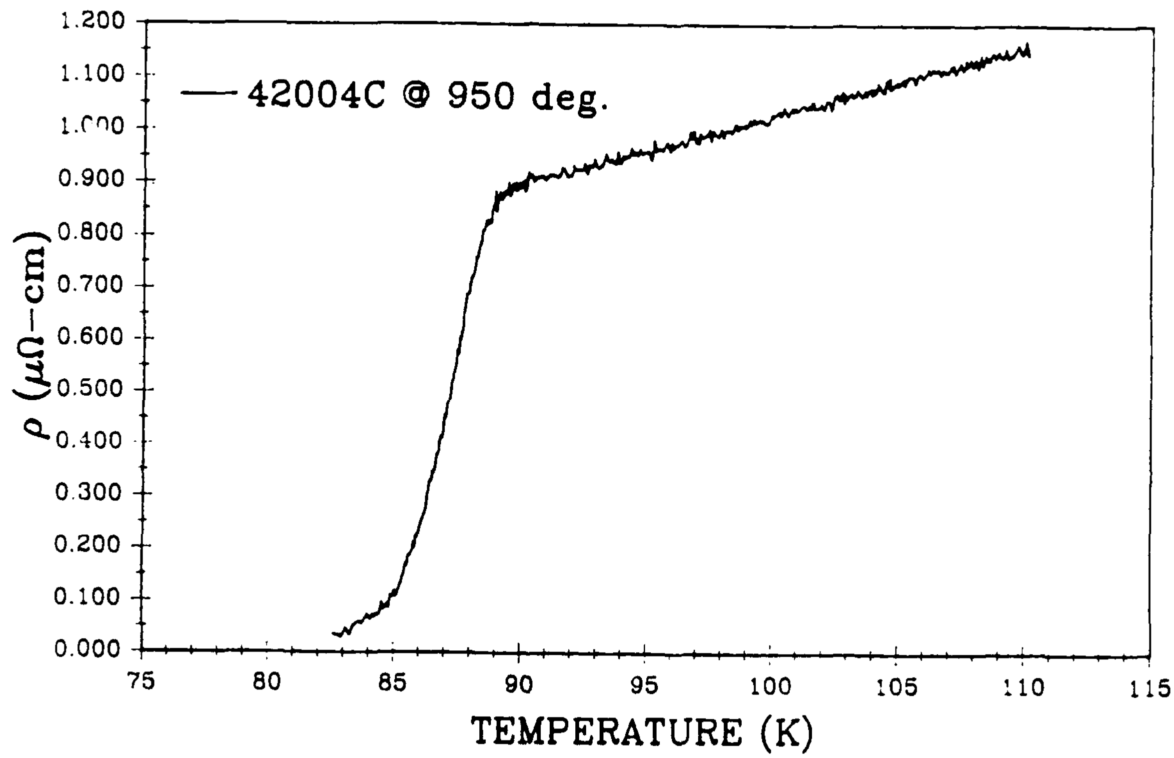


Figure 2.4.2 Resistive Transition in a Silver Clad Wire Continuously Sintered in Nitrogen at 945°C After a Post Anneal in Oxygen

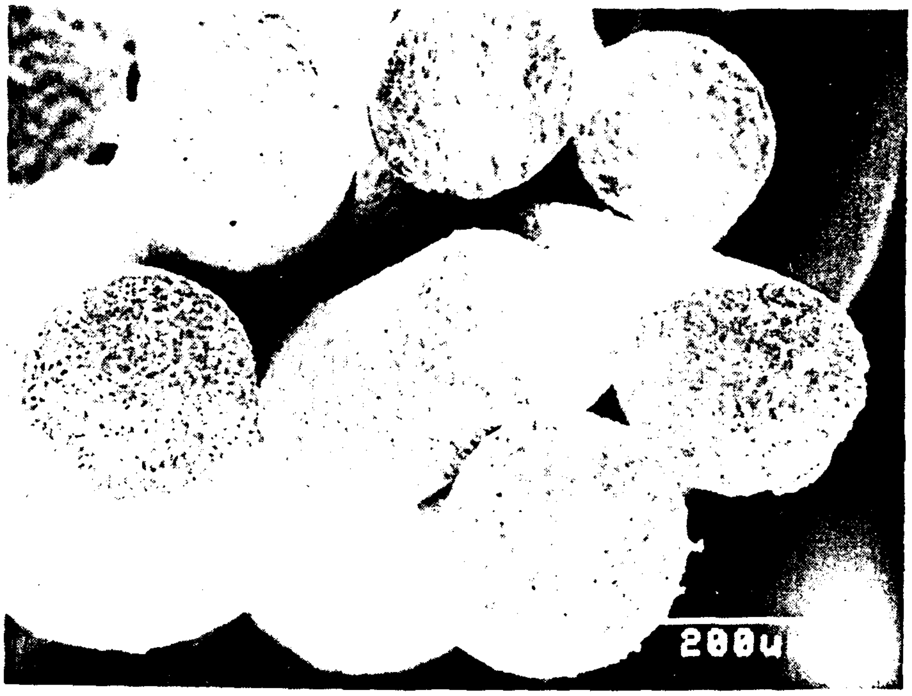


Figure 2.5.1 Seven Filament Braid of Y-123 After Rapid Thermal Anneal for One Second at 1025°C

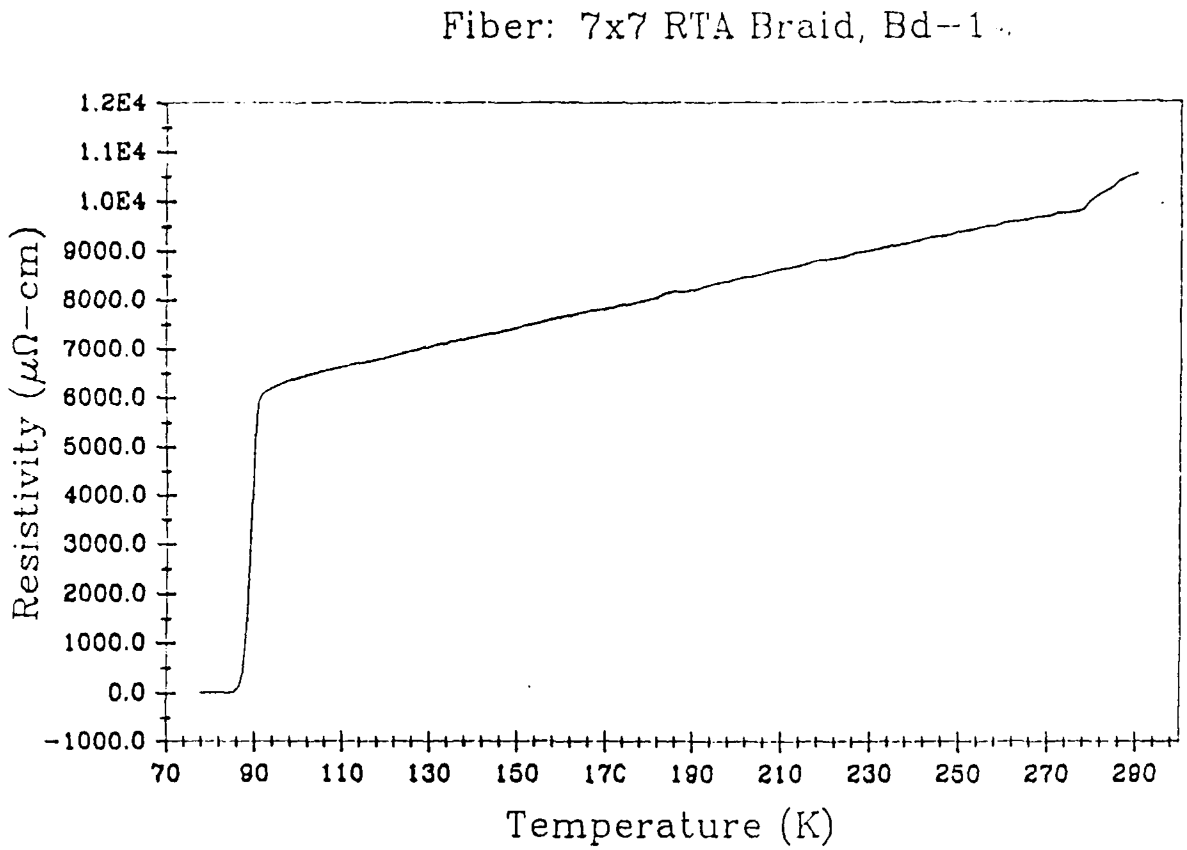


Figure 2.5.2 Resistive Transition in a Y-123 Braid After Rapid Thermal Anneal for One Second at 1025°C With No Post Anneal

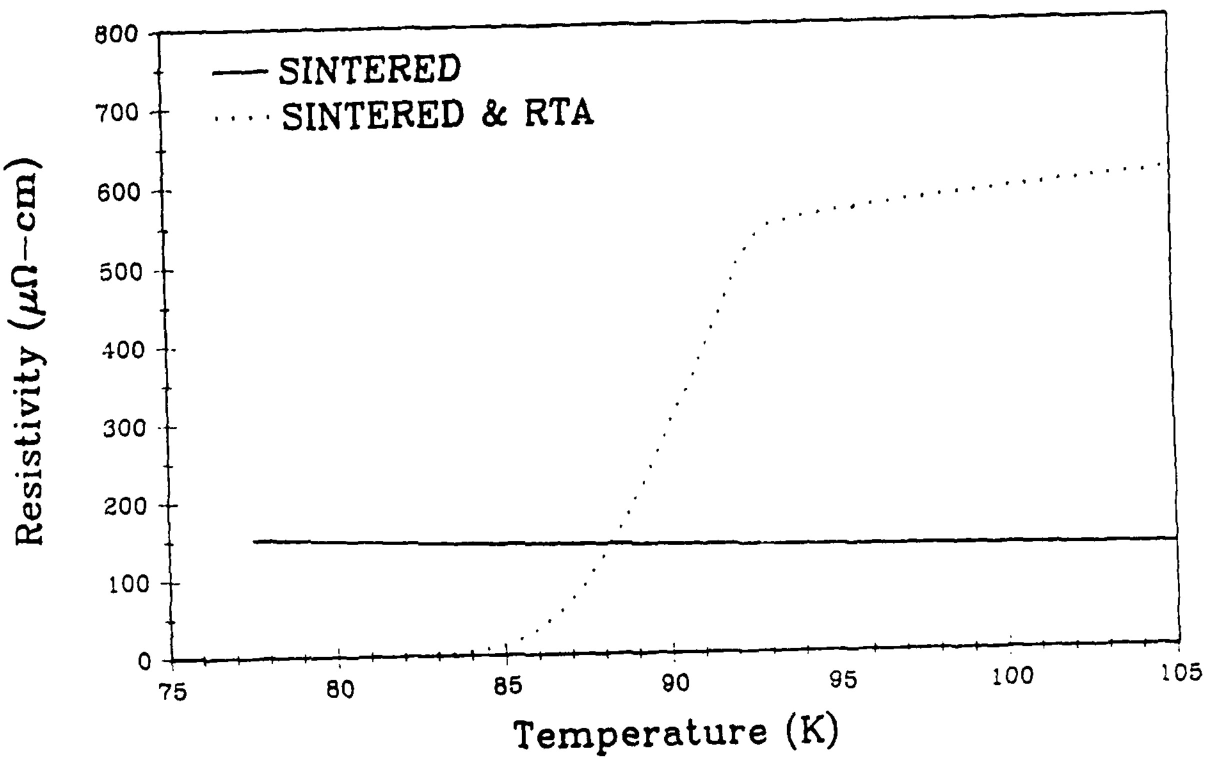


Figure 2.5.3 Resistive Transition in a Pre-Sintered Y-123 Filament after Four-Second 1025°C Rapid Thermal Anneal. Resistivity of A Similar Sintered Filament Shown for Comparison

SECTION 3

HIGH TEMPERATURE SUPERCONDUCTOR
MOTOR DESIGN AND FABRICATION

ALAN CRAPO AND JERRY LLOYD

EMERSON MOTOR COMPANY

3.1 Introduction

This quarter's superconductor motor work has concentrated on refining the design of the homopolar motor. Design improvements have been made since the last report, and brushes and springs have been tested in liquid nitrogen to make sure the brush system will work properly. Other motors to be built in the future are also discussed.

3.2 Homopolar Motor

We have made a few changes in the design this quarter to make the motor easier to build and improve performance.

The homopolar motor design of a quarter ago had a complex rotor. The rotor had radial ducts to bring power leads to the center of the rotor, and a hollow shaft to bring the leads out to slip rings on the shaft. The rotor design consisted of several pieces needing precision alignment. Also, connecting the power leads to the slip rings on the hollow shaft would have been very difficult. These items made the old design difficult to build.

The new design eliminates the radial ducts and hollow shaft with a one piece solid rotor. The radial ducts and hollow shaft on the old design

limited the flux produced by the HTSC coils at J_c values of 1300 A./cm^2. The solid rotor version in the new design will allow J_c values as high as 1700 A./cm^2 before we get any saturation effects. Figure 3.2.1 shows a flux plot of the new design with flux current, and torque directions identified. Figure 3.2.2 shows a side view cross section of the homopolar motor with various parts labeled. Figure 3.2.3 is an end view cross section showing the brush system layout.

The following list shows the parts required to build the motor, the quantity required, and where we are getting the part (our model shop or outside vendor).

PART	QUANTITY	SOURCE
A. Stator Assembly		
1. Stator center section	1	Model shop
2. Stator tube section	2	Model shop
3. Stator upper section	1	Model shop
4. Stator lower section	1	Model shop
5 Coil bobbin	2	Model shop
6. HTSC coils	2	CPS
7. Lead wires for field coil	2	In house
8. Bearings	2	In
9. Bolts	AR	Model shop
B. Rotor	1	Model shop
C. Brush Assembly		
1. Brushes	16	Morganite Corp.
2. Brush holders	16	Diamond Corp.

STEEL CORE HOMOPOLAR
MOTOR FLUX PLOT

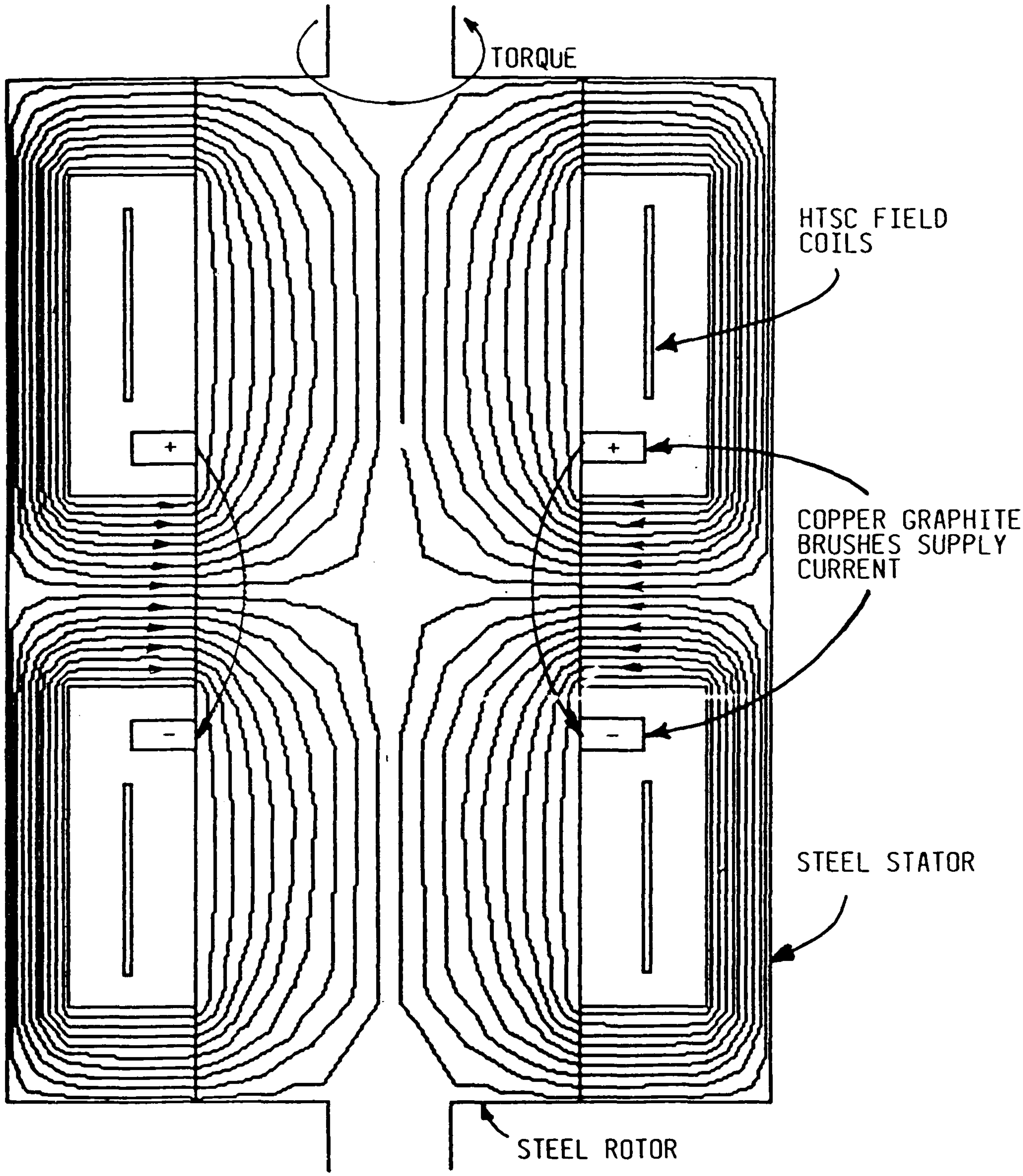


FIGURE 3.2.1

SUPERCONDUCTING HOMOPOLAR MOTOR DESIGN

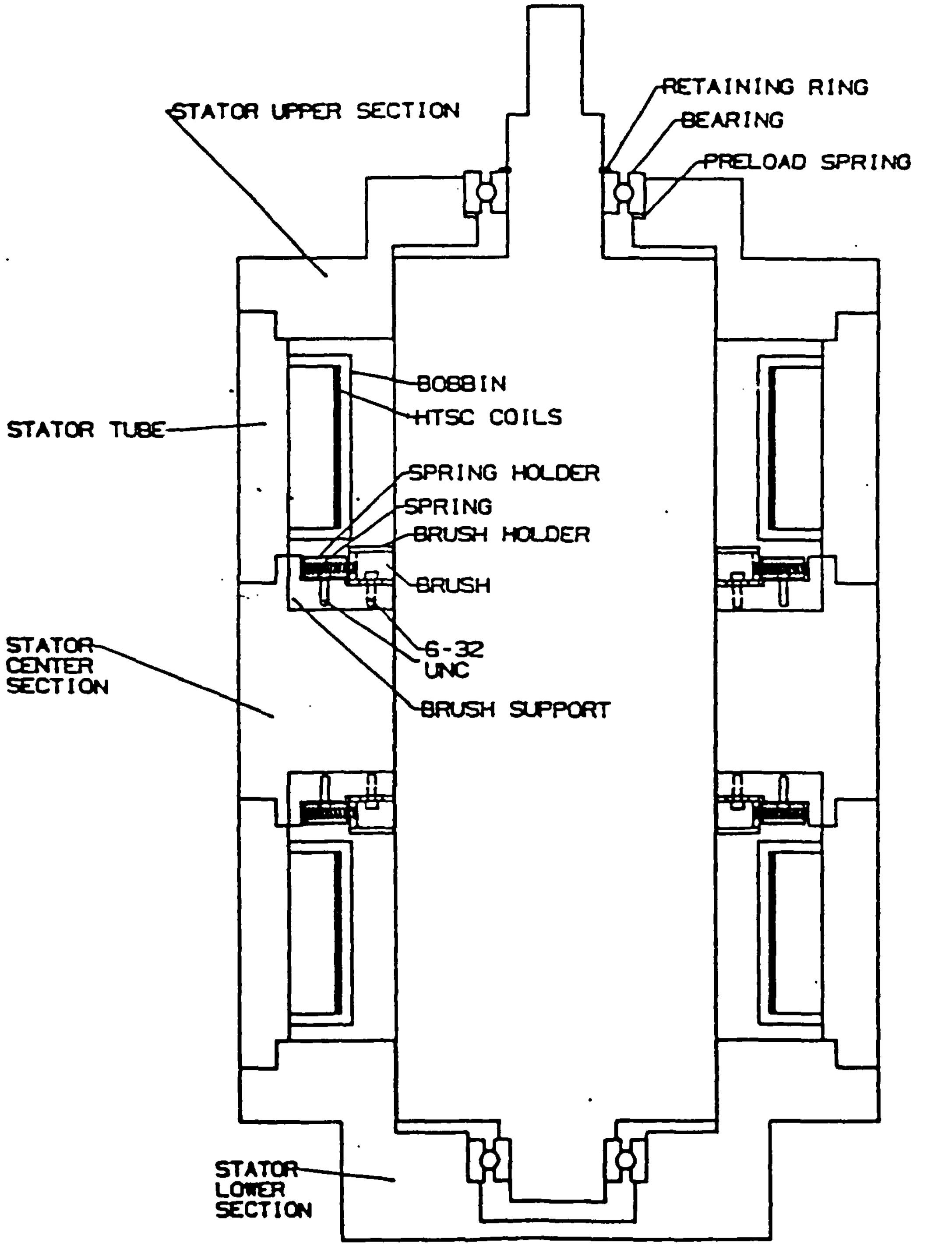


FIGURE 3.2.2

MOTOR CROSS-SECTION
(THROUGH BRUSH ASSEMBLY)

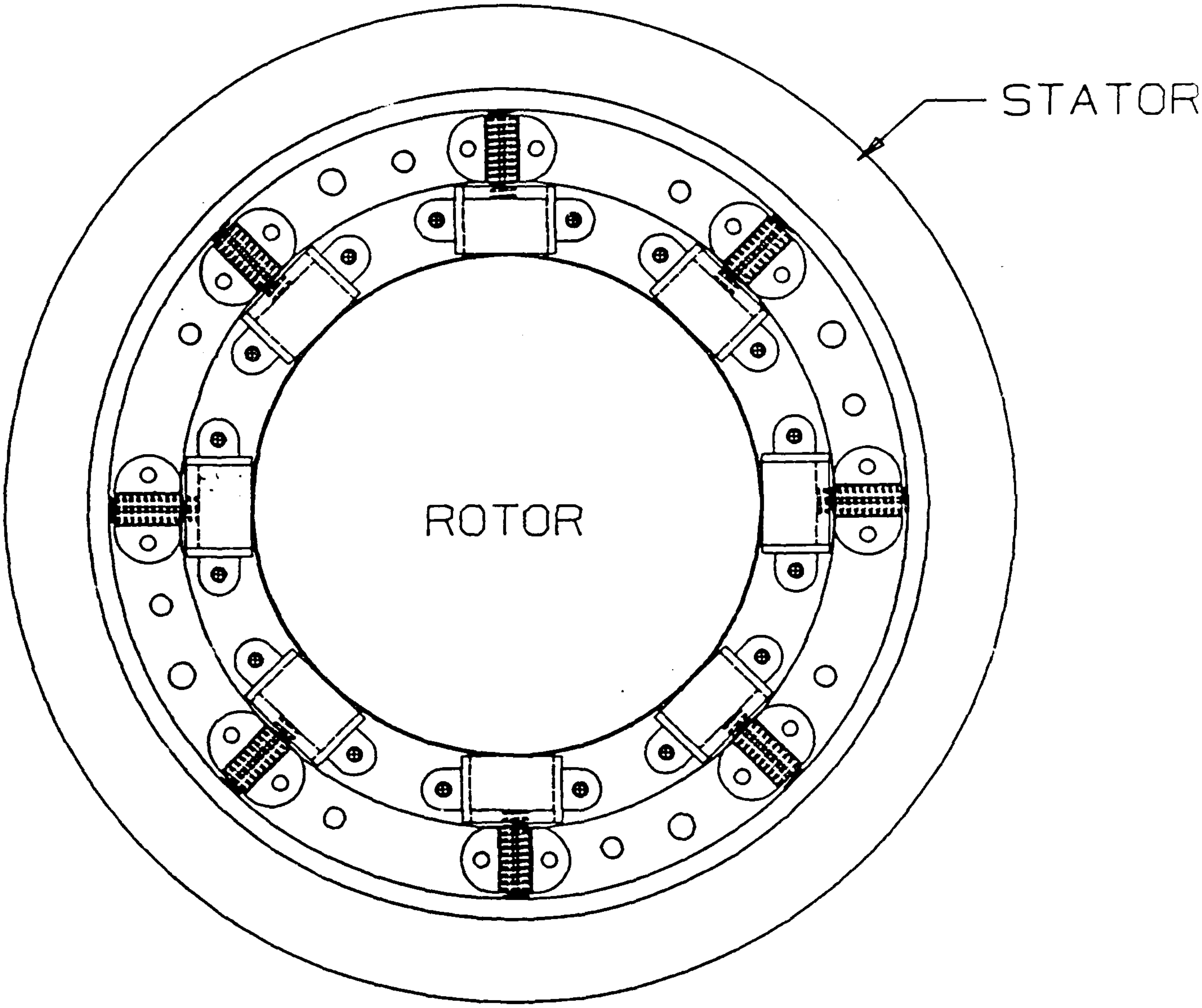


FIGURE 3.2.3

PART(cont)	QUANTITY	SOURCE
3. Spring holders	16	Diamond Corp.
4. 8 gauge leadwire	AR	In house
5. Bolts	AR	Model shop
6. Brush support ring	1	Model shop

We plan to assemble and begin testing next quarter.

We are working with Phelps Dodge Corporation and University of Wisconsin on the insulation for the HTSC wire. We will have a the insulation system established next quarter.

3.3 Testing

3.3.1 Spring constant tests

Since the bearing preload spring and brush springs will be at 77°K in the liquid nitrogen, the spring constant will be higher than at room temperature. Before we finalized the spring designs, we wanted to measure the change in spring constant. We also compared the Young's modulus of 304 stainless steel at room temperature and at 77°K.

	23°C (296°K)	77°K	% Change
Young's modulus	162 GPa	192 GPa	18.5%
Measured force at l_o	110 gf	120 gf	---
Measured force at l_o +0.5"	215 gf	245 gf	---
Measured force constant	82.68 gf/cm	98.43 gf/cm	19.0%

Both calculated and measured spring constant ratios indicated an increase of about 19% in dropping to 77°K. Spring designs now reflect the 19% increase in spring constant.

3.3.2 Brush Tests

We are setting up to do several brush tests in liquid nitrogen. We have two grades of brush to test, 65% copper, and 85% copper mixed with graphite. We will be testing these brushes on steel, copper, and brass slip rings at various speeds. We will be mostly looking for voltage drop versus current density in the brush.

Our motor will be much easier to build if we can use the steel slip ring. Our tests will compare the steel slip ring compared to copper and brass. We have some preliminary test data on the steel slip ring but we are doing it over to get more complete data. The first tests were done at 56 amperes/cm , There was a 0.5 Volt drop across two brushes in series with no signs of nitrogen boiling. This is equivalent to 1445 amperes in our homopolar motor.

Further testing will better documents our limits, but it looks like we should not have problems with the steel slip ring in our motor.

3.3.3 Homopolar tests

The first test on the HTSC homopolar motor will be on the field coil. They will be:

A. Voltage versus current or J_c measurement.

B. Total motor flux versus current.

C. Worst case flux density at the coil versus current.

We will want to evaluate what happens when part of the coil goes normal.

The second series of tests will be no load motor tests. We will test:

A. No load speed versus voltage.

B. Back EMF constant.

We will then test the motor under loaded conditions. We will test:

A. Speed versus torque for various current levels.

B. Voltage drop in brushes versus current.

We will then calculate the loss distribution and analyze the motor performance.

3.4 Other Motors

3.4.1. Induction Motor

The next motor to be built will be an induction motor with HTSC wire wound in the stator. The HTSC wire will be carrying AC current in an AC field. Like the homopolar motor, the induction motor will be tested while submerged in liquid nitrogen. Other than the HTSC stator winding, this motor will be the same as the conventional cryogenic induction motors made by U.S. Electrical Motors to run in liquid nitrogen.

Our plan is to build a 3 horsepower 1795 RPM motor that has a line current of 3.8 amperes RMS (5.4 amperes peak). We still need to do a finite element model to determine the worst case magnetic field at the wire. We need to work on making the wire so that it will bend around a 5 or 6 cm radius. We also need to protect the wire so it does not get damaged.

Testing the induction motor will give us a lot of useful information about the AC losses, and how the HTSC wire behaves with AC current in AC fields. Since most motors used in the United States today are induction motors, this is an important potential application for HTSC.

3.4.2. Next Generation Motors

Once we've been able to determine what we think the properties of HTSC wire will be a year from now, we will design a new motor that will be optimized around the improved characteristics. Our size goal for this motor will be in the 50 horsepower range. The motor will be built and then tested.

3.4.3. Future Motor Concepts

When HTSC wire properties for current density in a magnetic field, approach LTSC properties, many new high performance motors are possible. We will analyze the design possibilities and the potential applications, and define the design requirements to make these motors.

SECTION 4

GENERAL DISCUSSION AND SUMMARY

Progress this quarter was centered around achieving our first reel-to-reel continuous sintering of wire, and the production of prototype spools of monofilament wire. In support of this activity, and in addition to it, there was significant progress in all major process steps for wire manufacture, initial explorations of melt texturing, and important developments in HTSC motor design.

Powder production facilities were upgraded to improve the jet milling facility, and to allow us to process Y-123 powder in water- and carbon dioxide-free gas. The Y-123 production lot size was increased to 10 kilograms. Quality control standards and powder specifications were improved.

We routinely produce spools of 125-micron diameter green fiber (50 vol% Y-123) in lengths up to 1000 feet as feedstock for sintering and cladding. We determined that it was possible to increase the Y-123 powder loading in the green fiber from 50 vol% to 60 vol% without sacrificing green fiber properties. The higher solids loading is expected to improve burnout and sintering behavior. Efforts continue to improve the fiber spinning process and determine the influence of process variables on green fiber quality. Efforts continue on evaluation of alternative polymer systems. Polyethylene-based systems remain the most useful, although one attractive alternative was identified.

Although the primary emphasis was on monofilament wire, development continued on producing the multifilamentary braids which will be needed

for the ribbon wire. Eight filament braids up to 25 feet long have been produced with 125-micron fiber.

Monofilament wire development focused on co-fired silver coated green fibers. Reformulation of the silver coating material has significantly improved the thickness, adherence, and uniformity of the cladding. Coating procedures were developed to produce fiber for sintering with very smooth silver coatings. Silver thickness between 20 and 100 microns can be easily produced, allowing variation of the metal/superconductor ratio between 1 and 8. Continuous coated fiber has been made at lengths up to 150 feet. Conditions for successful co-firing of the fiber with Ag and Ag/Pd coatings are being determined.

The major emphasis this quarter has been on continuous co-firing of silver alloy coated green fiber. Pay-out and take-up spoolers were installed in the sintering facility for reel-to-reel operation. The current process is limited by length of the heated zones, so the fiber must pass through the furnace several times for burnout, presintering, and sintering with intermediate spooling. Furnace modifications are being designed.

Continuously sintered Ag-clad wire has been produced and collected on a spool. The wire has a resistive transition in the as-sintered state and becomes fully superconducting after an oxygen anneal. Optimization of the time-temperature-atmosphere firing profile is underway, although presently constrained by intermediate spoolings. Very slow belt speeds are now being used to avoid defects associated with binder burnout. Slow speed and the need for multiple passes limits the length of sintered wire to about 25 feet. Localized defects in the silver cladding create weak spots leading to breaks. The longest length without a break has been 19 feet.

The ribbon conductor cladding module for reflow bonding has been completed. Evaluation is postponed by emphasis on the co-fired monofilamentary wire.

Electrical measurements activity this quarter was primarily for routine characterization of transport properties of sintered fibers and wires.

Emerson Electric completed the design for the first generation HTSC motor, a drum DC homopolar with an iron magnetic circuit. Several design improvements were made. Purchased parts, components, and equipment have been ordered. Major motor parts are being machined. The performance of the brush system has been tested at liquid nitrogen temperature. The motor will be assembled and tested with copper coils in the following quarters.

The HTSC coils will be wound on a 10-inch diameter bobbin. Two coils are required with 4600 turns of monofilament wire, for a total wire length of 3000 meters per coil.

Research on a melt texturing process began, with emphasis on a method which could ultimately be incorporated in the fiber process for wire manufacture. Laser floating zone crystal growth, rapid thermal processing, and quenched powder methods are being explored.

REFERENCES

1. K. Sawano, M. Morita, K. Miyatomo, K. Doi, A.Hayashi, M. Murakami, and S. Matsuda, "Effects of Synthesis Conditions on Microstructure of Y-123 Superconductor by Partial Melting Process", to appear in Seramikkusu Ronbunshi, 1989

2. M. Murakami, M. Morita, K. Miyamoto, submitted to Japan J. Applied Physics (1989)

ATTACHMENT I

REPORT SUMMARY

COMPOSITE CERAMIC SUPERCONDUCTING WIRES FOR ELECTRIC MOTOR APPLICATIONS

Fifth Quarterly Report on
Contract Number N00014-88-C-0512

October 10, 1989

John W. Halloran, Ceramics Process Systems Corporation,
Milford, MA 01757

This report describes progress on developing Y-123 wire for an HTSC motor. The wire development activity includes synthesis of Y-123 powder, spinning polymer-containing "green fiber", heat treating the fiber to produce metallized superconducting filaments, and characterizing the electrical properties of the filaments.

Powder production facilities were upgraded and production was increased to 10 kilograms. Spools of 125-micron diameter green fiber (50 vol% Y-123) are routinely produced in lengths up to 1000 feet for sintering and cladding. Powder loading in the green fiber was increased to 60 vol%. Eight filament braids up to 25 feet long have been produced with 125-micron fiber. Efforts continue on evaluation of alternative polymer systems.

Wire development is focused on co-fired silver coated monofilaments. The silver coating material now has significantly better thickness and uniformity for metal/superconductor ratios of 1 to 8. Continuous coated fiber has been made at lengths up to 150 feet.

The major emphasis has been on continuous co-firing of silver alloy coated green fiber in a reel-to-reel operation. The current process is limited by length of the heated zones, so the fiber must pass through the furnace several times for burnout, presintering, and sintering with intermediate spooling. Furnace modifications are being designed. Continuously sintered Ag-clad wire has been produced and collected on a spool. The wire has a resistive transition in the as-sintered state and becomes fully superconducting after an oxygen anneal. Localized defects in the silver cladding create weak spots leading to breaks. The longest length without a break has been 19 feet.

Design was completed for the first generation HTSC motor, a drum DC homopolar with an iron magnetic circuit. Purchased parts, components, and equipment have been ordered. Major motor parts are being machined. Tests proved the performance of a brush system at liquid nitrogen temperature. The motor will be assembled and tested with copper coils in the following quarters.

ATTACHMENT II

ARPA ORDER NUMBER: 9525

PROGRAM CODE NUMBER: 7737

CONTRACTOR: Ceramics Process Systems Corporation
155 Fortune Boulevard
Milford, MA 01757

CONTRACT NUMBER: N00014-88-C-0512

CONTRACT EFFECTIVE DATE: 30 JUNE 1988

CONTRACT EXPIRATION DATE: 31 MARCH 1991

SHORT TITLE OF WORK: High Temperature Superconducting Wire and Motor

PRINCIPAL INVESTIGATOR: John W. Halloran
(508) 634-3422

This report describes progress on developing Y-123 wire for an HTSC motor. The wire development activity includes synthesis of Y-123 powder, spinning polymer-containing "green fiber", heat treating the fiber to produce metallized superconducting filaments, and characterizing the electrical properties of the filaments.

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ATTACHMENT III

ARPA ORDER NUMBER: 9525 PROGRAM CODE NUMBER: 7737

CONTRACTOR: Ceramic Process Systems Corporation
155 Fortune Boulevard
Milford, MA 01757

CONTRACT NUMBER: N00014-88-C-0512 CONTRACT AMOUNT: $ 5,509,387.00

EFFECTIVE DATE OF CONTRACT: 30 JUNE 1988

EXPIRATION DATE OF CONTRACT: 31 MARCH 1991

PRINCIPAL INVESTIGATOR: John W. Halloran

TELEPHONE NUMBER: (508) 634-3422

SHORT TITLE OF WORK: High Temperature Superconducting Wire and Motor

REPORTING PERIOD: 1 JULY 1989 through 30 SEPTEMBER 1989

DESCRIPTION OF PROGRESS

Powder production facilities were upgraded and production was increased to 10 kilograms. Spools of 125-micron diameter green fiber (50 vol% Y-123) are routinely produced in lengths up to 1000 feet for sintering and cladding. Powder loading in the green fiber was increased to 60 vol%. Eight filament braids up to 25 feet long have been produced with 125-micron fiber. Efforts continue on evaluation of alternative polymer systems.

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<u>SUMMARY OF SUBSTANTIVE INFORMATION DERIVED FROM SPECIAL EVENTS</u>

Project members attended the several conferences on high temperature superconductivity to keep abreast of current developments.

<u>CHANGE IN KEY PERSONNEL</u>

No change

<u>PROBLEMS ENCOUNTERED AND/OR ANTICIPATED</u>

None

<u>ACTION REQUIRED BY THE GOVERNMENT</u>

None

<u>FISCAL STATUS (SEPTEMBER 30,1989)</u>

1) <u>CUMULATIVE AMOUNT CURRENTLY RECEIVED ON CONTRACT</u> . . . . $ 1,900,440

2) <u>CUMULATIVE EXPENDITURES AND COMMITTMENTS TO DATE</u>........................... $ 2,023,996

3) <u>ADDITIONAL FUNDS REQUIRED TO COMPLETE WORK</u>

THROUGH FY 1991......................... $ 2,204,000